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(54) Title: DETERGENT COMPOSITIONS

(57) Abstract

A non-bleaching laundry detergent composition comprises surfactant, builder, and optionally other non-bleach detergent ingredients, and also contains from 0.05 to 2.5 wt.% of iminodisuccinate (IDS) or hydroxyiminodisuccinate (HIDS). The composition provides improved maintenance and/or restoration of colour fidelity during the wash, especially at low wash pH. The IDS or HIDS is also an effective chlorine scavenger, reducing the in-wash fading of chlorine-sensitive dyes. In addition, the incorporation of the IDS or HIDS improves the stain removal performance of the composition.

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DETERGENT COMPOSITIONS

TECHNICAL FIELD

The present invention relates to laundry detergent compositions suitable for washing both white and coloured fabrics. The compositions of the invention, which contain a sequestrant, iminodisuccinate or hydroxyiminodisuccinate, in controlled amounts, have been found to give improved maintenance and/or restoration of colour fidelity during the wash. The presence of the sequestrant also reduces dye fading due to chlorine in water, and in addition provides improved stain removal.

BACKGROUND

Certain colours used in the textile industry have the tendency to degrade during the laundry process. Coloured articles may become darker or the colour may change as a result of laundering, thus changing the appearance and shortening the useful life of the articles. White articles may also become discoloured, for example, yellowed, with time.

It has now been found that new fabrics may be protected against such colour degradation, and the colour fidelity of previously washed fabrics may be restored (ie the degradation reversed), if the fabrics are laundered using non-bleaching detergent compositions containing controlled amounts of iminodisuccinic acid or its water-soluble salts.

The benefits are especially marked in the washing of coloured cotton at low pH.

Fading of coloured fabrics in the wash because of the presence of sodium hypochlorite and other chlorine compounds in water is also a known problem. It has been found that iminodisuccinic acid and its salts are also effective chlorine scavengers and the inclusion of these compounds also mitigates this problem.

Additionally, the inclusion of these compounds also gives improved removal of certain stains, especially red mud.

PRIOR ART

Iminodisuccinate (IDS) is known as a detergency builder and, in bleaching detergent compositions, as a stabiliser for peroxy bleach precursors.

US 3 697 453 (Pfizer) discloses detergent compositions having a pH of from 9 to 12, containing iminodisuccinate as a detergency builder, used together with detergent surfactant in a weight ratio of 0.25:1 to 10:1. IDS as a detergency builder is also disclosed in EP 757 094A (Bayer). IDS and hydroxyiminodisuccinate (HIDS) are disclosed in JP 09 110 813A (Nippon Shokubai) and JP 09 104 897A (Nippon Shokubai).

EP 509 382A (W.R Grace & Co/Hamphire Chemical Corporation) discloses a bleaching detergent composition comprising a bleaching agent and a bleach stabiliser of defined formula.

which includes IDS. The use of IDS as a bleach stabiliser is also disclosed in WO 97/20907A (Procter & Gamble).

JP 09 249 895A (Lion) and JP 09 310 097A (Lion) disclose detergent compositions containing 3 to 20 wt% IDS or HIDS to improve the stability or fabric substantivity of fluorescers (optical brighteners).

The use of IDS as a processing aid for detergent powders and detergent powder ingredients is disclosed in JP 09 100 497A (Lion) and JP 09 279 188A (Lion).

WO 98 38276A (Procter & Gamble) discloses laundry detergent compositions containing 0.1-50 wt% of a colour care agent. The colour care agents are amines substituted with, for example, hydroxyalkyl groups. The preferred material is N,N,N',N'-tetrakis-(2-hydroxypropyl)ethylenediamine.

WO 91 17234A (Procter & Gamble) discloses low-pH granular laundry detergent compositions containing chlorine scavengers which minimise the fading of pH-sensitive and chlorine-sensitive fabric dyes during laundering. Preferred chlorine scavengers are ammonium salts, for example, ammonium sulphate.

DEFINITION OF THE INVENTION

The present invention accordingly provides a non-bleaching laundry detergent composition providing improved maintenance and/or restoration of colour fidelity during the wash, the composition comprising surfactant, builder, and optionally other non-bleach detergent ingredients, and also containing from 0.05 to 2.5 wt% of a compound of the formula (1):

wherein Y is H or OH and X is H or a solubilising cation.

A further subject of the invention is a process for laundering white or coloured textile fabrics while maintaining and/or restoring the colour fidelity of the fabrics, which process comprises laundering the fabrics by hand or machine in a wash liquor containing a detergent composition as defined previously.

A further subject of the invention is a method of protecting new white or coloured textile fabrics from colour degradation on laundering, which comprises laundering the fabrics by hand or machine in a wash liquor containing a detergent composition as defined above.

A further subject of the invention is a method of restoring colour fidelity in white or coloured textile fabrics that have been laundered, which comprises laundering the fabrics by hand or machine in a wash liquor containing a detergent composition as defined above.

A further subject of the invention is a method of removing stains from textile fabrics, which comprises laundering the fabrics by hand or machine in a wash liquor containing a detergent composition as defined above.

A further subject of the invention is the use of the compound of the formula I above in an amount of 0.05 to 2.5 wt% in a laundry detergent composition to protect new white or coloured textile fabrics from colour degradation on laundering, or to restore colour fidelity in white or coloured textile fabrics that have been laundered.

The term "colour fidelity" is used herein to include both the true colour of coloured fabrics and the whiteness of white fabrics.

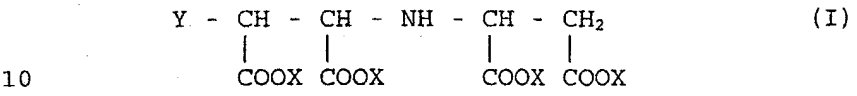
A further subject of the invention is the use of a compound of the formula I above, in an amount of from 0.05 to 2.5 wt%, in a laundry detergent composition as a chlorine scavenger to prevent the fading of chlorine-sensitive dyes on coloured textile fabrics during laundering.

A further subject of the invention is the use of a compound of the formula I above, in an amount of from 0.05 to 2.5 wt%, in a laundry detergent composition to improve its stain removal performance.

DETAILED DESCRIPTION OF THE INVENTION

The compound of formula I

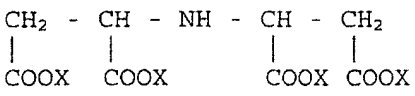
The detergent compositions of the invention contain, as an essential ingredient, a compound of the formula I:



(I)

wherein Y is H or OH, preferably H; and X is H or a solubilising cation, preferably a sodium ion.

If Y is a hydrogen atom, the formula I represents iminodisuccinic acid or a water-soluble salt thereof. Iminodisuccinic acid, also known as N-(1,2-carboxyethyl)D,L-aspartic acid, has the formula (Ia) wherein X = H:

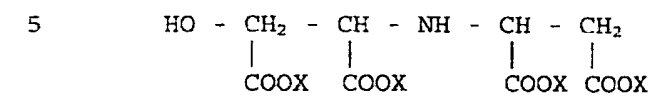


(Ia)

In the following description, the abbreviation "IDS" will be used to denote this material whether in acid or salt form. IDS is commercially available from Bayer AG, Leverkusen, Germany, and from Nippon Shokubai KK, Japan.

If Y is a hydroxyl group, the formula I represents hydroxyiminodisuccinic acid or a water-soluble salt thereof.

Hydroxyiminodisuccinic acid has the formula (Ib) wherein X = H:



(Ib)

In the following description, the abbreviation "HIDS" will be used to denote this material whether in acid or salt form. HIDS is commercially available from Nippon Shokubai KK, Japan.

For the purposes of the present invention, the IDS or HIDS may be, and preferably is, in the form of a salt, ie X in the formula I is a stable solubilising cation, preferably an alkali metal cation, more preferably sodium.

In the laundry detergent compositions of the invention, IDS or HIDS is present in an amount of from 0.05 to 2.5 wt%, preferably from 0.2 to 2.5 wt%, more preferably from 0.3 to 1.5 wt% and most preferably from 0.5 to 1.0 wt%. The lower levels appear to provide the greatest benefit and no additional benefit is observed if higher amounts, greater than 2.5 wt%, are used.

The preferred material is IDS, most preferably in sodium salt form.

Detergent compositions

The composition of the invention also contains other conventional detergent ingredients, other than bleaching ingredients. Essential ingredients are surfactants (detergent-active compounds) and detergency builders, and other non-bleach ingredients may optionally be present.

A preferred detergent composition according to the invention comprises:

(a) from 5 to 60 wt% of one or more detergent surfactants,

(b) from 10 to 80 wt% of one or more detergency builders,

(c) from 0.05 to 2.5 wt%, preferably from 0.2 to 2.5 wt%, more preferably from 0.3 to 1.5 wt% and most preferably from 0.5 to 1.0 wt%, of IDS or HIDS,

(d) optionally other non-bleach detergent ingredients to 100 wt%.

The detergent compositions of the invention may be of any physical form.

Surfactants (detergent-active compounds)

The detergent compositions will contain, as essential ingredients, one or more detergent active compounds (surfactants) which may be chosen from soap and non-soap

anionic, cationic, nonionic, amphoteric and zwitterionic detergent active compounds, and mixtures thereof. Many suitable detergent active compounds are available and are fully described in the literature, for example, in "Surface-Active Agents and Detergents", Volumes I and II, by Schwartz, Perry and Berch.

The preferred detergent active compounds that can be used are soaps and synthetic non-soap anionic and nonionic compounds.

Anionic surfactants are well-known to those skilled in the art. Examples include alkylbenzene sulphonates, particularly linear alkylbenzene sulphonates having an alkyl chain length of C_8-C_{15}; primary and secondary alkylsulphates, particularly C_8-C_{15} primary alkyl sulphates; alkyl ether sulphates; olefin sulphonates; alkyl xylene sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates. Sodium salts are generally preferred.

Nonionic surfactants that may be used include the primary and secondary alcohol ethoxylates, especially the C_8-C_{20} aliphatic alcohols ethoxylated with an average of from 1 to 20 moles of ethylene oxide per mole of alcohol, and more especially the C_{10}-C_{15} primary and secondary aliphatic alcohols ethoxylated with an average of from 1 to 10 moles of ethylene oxide per mole of alcohol. Non-ethoxylated nonionic surfactants include alkylpolyglycosides, glycerol monoethers, and polyhydroxyamides (glucamide).

Cationic surfactants that may be used include quaternary ammonium salts of the general formula $R_1R_2R_3R_4N^+ X^-$ wherein the R groups are long or short hydrocarbyl chains, typically

alkyl, hydroxyalkyl or ethoxylated alkyl groups, and X is a solubilising cation (for example, compounds in which R_1 is a C_8-C_{22} alkyl group, preferably a C_8-C_{10} or C_{12}-C_{14} alkyl group, R_2 is a methyl group, and R_3 and R_4, which may be the same or different, are methyl or hydroxyethyl groups); and cationic esters (for example, choline esters).

In an especially preferred cationic surfactant of the general formula $R_1R_2R_3R_4N^+ X^-$, R_1 represents a C_8-C_{10} or C_{12}-C_{14} alkyl group, R_2 and R_3 represent methyl groups, and R_4 presents a hydroxyethyl group.

Amphoteric surfactants, for example, amine oxides, and zwitterionic surfactants, for example, betaines, may also be present.

Preferably, the quantity of anionic surfactant is in the range of from 5 to 50% by weight of the total composition. More preferably, the quantity of anionic surfactant is in the range of from 8 to 35% by weight.

Nonionic surfactant, if present, is preferably used in an amount within the range of from 1 to 20% by weight.

The total amount of surfactant present is preferably within the range of from 5 to 60 wt%.

Detergency builders

The compositions may suitably contain from 10 to 80%, preferably from 15 to 70% by weight, of detergency builder.

Preferably, the quantity of builder is in the range of from 15 to 50% by weight.

The detergent compositions may contain as builder a crystalline aluminosilicate, preferably an alkali metal aluminosilicate, more preferably a sodium aluminosilicate (zeolite).

The zeolite used as a builder may be the commercially available zeolite A (zeolite 4A) now widely used in laundry detergent powders. Alternatively, the zeolite may be maximum aluminium zeolite P (zeolite MAP) as described and claimed in EP 384 070B (Unilever), and commercially available as Doucil (Trade Mark) A24 from Crosfield Chemicals Ltd, UK. Zeolite MAP is defined as an alkali metal aluminosilicate of zeolite P type having a silicon to aluminium ratio not exceeding 1.33, preferably within the range of from 0.90 to 1.33, preferably within the range of from 0.90 to 1.20.

Especially preferred is zeolite MAP having a silicon to aluminium ratio not exceeding 1.07, more preferably about 1.00. The particle size of the zeolite is not critical. Zeolite A or zeolite MAP of any suitable particle size may be used.

Also preferred according to the present invention are phosphate builders, especially sodium tripolyphosphate. This may be used in combination with sodium orthophosphate, and/or sodium pyrophosphate.

Other inorganic builders that may be present additionally or alternatively include sodium carbonate, layered silicate, amorphous aluminosilicates.

Organic builders that may be present include polycarboxylate polymers such as polyacrylates and acrylic/maleic copolymers, polyaspartates, monomeric polycarboxylates such as citrates, gluconates, oxydisuccinates, glycerol mono-di- and trisuccinates, carboxymethyloxysuccinates, carboxymethyloxymalonates, dipicolinates, hydroxyethyliminodiacetates, alkyl- and alkenylmalonates and succinates, and sulphonated fatty acid salts.

Organic builders may be used in minor amounts as supplements to inorganic builders such as phosphates and zeolites. Especially preferred supplementary organic builders are citrates, suitably used in amounts of from 5 to 30 wt %, preferably from 10 to 25 wt %; and acrylic polymers, more especially acrylic/maleic copolymers, suitably used in amounts of from 0.5 to 15 wt %, preferably from 1 to 10 wt%.

Builders, both inorganic and organic, are preferably present in alkali metal salt, especially sodium salt, form.

Other ingredients

The detergent compositions may also contain one or more enzymes. Suitable enzymes include the proteases, amylases, cellulases, oxidases, peroxidases and lipases usable for incorporation in detergent compositions.

Preferred proteolytic enzymes (proteases) are catalytically active protein materials which degrade or alter protein types of stains when present as in fabric stains in a hydrolysis reaction. They may be of any suitable origin, such as vegetable, animal, bacterial or yeast origin.

Proteolytic enzymes or proteases of various qualities and origins and having activity in various pH ranges of from 4-12 are available. Proteases of both high and low isoelectric point are suitable.

Other enzymes that may suitably be present include lipases, amylases, and cellulases including high-activity cellulases such as "Carezyme").

In particulate detergent compositions, detergency enzymes are commonly employed in granular form in amounts of from about 0.1 to about 3.0 wt%. However, any suitable physical form of enzyme may be used in any effective amount.

Antiredeposition agents, for example cellulose esters and ethers, for example sodium carboxymethyl cellulose, may also be present.

The compositions may also contain soil release polymers, for example sulphonated and unsulphonated PET/POET polymers, both end-capped and non-end-capped, and polyethylene glycol/polyvinyl alcohol graft copolymers such as Sokolan (Trade Mark) HP22.

Especially preferred soil release polymers are the sulphonated non-end-capped polyesters described and claimed in WO 95 32997A (Rhodia Chimie).

Other ingredients that may be present include solvents, hydrotropes, fluorescers, photobleaches, foam boosters or foam controllers (antifoams) as appropriate, sodium carbonate, sodium bicarbonate, sodium silicate, sodium

sulphate, calcium chloride, other [illegible] salts, fabric conditioning compounds, and perfumes.

Product form

As previously indicated, the compositions of the invention may be of any suitable physical form, for example particulates (powders, granules, tablets), liquids, pastes, gels or bars.

According to one especially preferred embodiment of the invention, the detergent composition is in particulate form.

If necessary, the IDS may be incorporated in particulate compositions in the form of granules containing an inert carrier material.

Compositions in powder form may be of any bulk density and may be prepared by spray-drying, non-tower granulation, or any combination of these techniques.

According to another especially preferred embodiment of the invention, the detergent composition is in liquid form.

Liquid detergent compositions may be prepared by admixing the essential and optional ingredients in any desired order to provide compositions containing the ingredients in the the requisite concentrations.

The colour care benefit

The colour benefit associated with the compositions of the invention is twofold: protection (maintenance) of the colour or whiteness of new fabrics, and restoration (recovery) of the colour or whiteness of previously washed fabrics.

New fabrics, when washed with the compositions of the invention, show reduced colour degradation as compared with fabrics washed in control formulations without IDS. The fabrics maintain a new appearance even after multiple washing. This protection or maintenance benefit has been found to apply both to white and to coloured fabrics.

Previously washed fabrics which have already undergone colour degradation show significant improvements in colour fidelity when washed using compositions according to the invention. Thus colour damage that has already occurred can be reversed and the new appearance of the fabrics restored.

It is believed that a cause of the colour degradation resulting from the laundry process may be heavy metal ions, especially iron but also copper, zinc and manganese: these may originate from the water or water pipes, from washing machine parts, from the detergent composition used, or from the soil present on the fabric.

Without wishing to be bound by theory, it is believed that the colour care benefit obtained according to the present invention may be attributed to the sequestration by the IDS of any heavy metal ions present in the wash liquor.

However, it is not known why IDS should be especially effective in this regard. In particular, it is surprising that IDS should be significantly more effective that the more common detergent and highly effective sequestrant, nitrilotriacetate (NTA).

pH of composition

According to a preferred embodiment of the invention, the composition has a 0.25% aqueous solution pH (in demineralised water at 30°C) not exceeding 10.5, preferably not exceeding 10.0. The preferred pH range is from 8.0 to 10.5, more preferably from 8.5 to 10.0.

The colour care benefits of the invention have been found to be especially marked under low-pH conditions.

The wash process

As indicated previously, a further subject of the invention is a process in which white or coloured fabrics are laundered using the composition of the invention. According to a preferred embodiment of the invention, the process is carried out in a wash liquor having a pH not exceeding 10.5, more preferably not exceeding 10.0.

It has been found that the colour care benefits of the invention, both protection and recovery, are especially marked when the wash liquor pH is low.

Particularly good results are obtained at pH values of from 8.0 to 10.5, preferably from 8.5 to 10.0.

Whilst the invention is also applicable to the machine wash, the two preferred conditions previously mentioned - very low pH, and low sequestrant level - have especial relevance to the handwash as carried out, for example, in South East Asia. In that region, it is a common habit to presoak or prerinse the fabrics in water, without detergent, to remove gross soiling. At this stage the wash liquor pH may be as low as 6.0 to 7.0. The wet fabrics are then immersed in the main wash liquor (detergent solution). The additional water imported into the wash liquor from the wet fabrics has the dual effect of diluting the wash liquor (and thus lowering the sequestrant level) and lowering the pH.

Under these conditions, especially significant colour care benefits according to the present invention have been observed.

The chlorine scavenging benefit

The incorporation of IDS has also been found to reduce the fading upon laundering of chlorine-sensitive dyes. This fading occurs due to the presence of sodium hypochlorite, which is routinely put into supply water for hygiene purposes: levels of 0.5 ppm are typical. This causes fading of dyes of a wide range of colours. The incorporation of IDS in accordance with the present invention can significantly reduce the amount of fading attributable to chlorine in the wash water.

The stain removal benefit

A further benefit for the incorporation of IDS has unexpectedly been observed: a significant improval in the removal of certain highly coloured stains, notably red mud. Better removal of blood and tea stains is also observed.

EXAMPLES

The invention will now be illustrated in further detail by means of the following Examples, in which parts and percentages are by weight unless otherwise stated. Examples designated with a number illustrate the invention, while examples designated with a letter are comparative.

EXAMPLE 1, COMPARATIVE EXAMPLES A TO G

Protection of new coloured fabrics from colour degradation by Cu^{2+} ions using sequestrants

In these experiments, the protective effect of IDS on cotton fabrics dyed with Direct Red 80 against colour degradation in the presence of copper ions was demonstrated and compared with other sequestrants.

The sequestrants used were as follows:

IDS: iminodisuccinate, tetrasodium salt, ex Bayer

NTA: nitrilotriacetate, trisodium salt, ex Aldrich

EDTMP: ethylenediaminetetramethylene phosphonate, calcium salt (Dequest (Trade Mark) 2047 ex Monsanto)

EDDS: ethylenediamine disuccinate, tetrasodium salt (Octaquest (Trade Mark) E30 ex Associated Octel)

STP: sodium tripolyphosphate (Polyphos technical grade ex Thai Polyphosphate and Chemicals)

Magnesium silicate: Macrosorb (Trade Mark) MS33 ex Crosfield Chemicals, UK.

The fabrics were washed in demineralised water containing copper ions (0.5 ppm Cu^{2+} ex $CuCl_2$) at a liquor to cloth ratio of 200:1 in tergotometers for 30 minutes at 30°C at 90 rpm.

Experiments were carried out at two different pH values, 9.5 and 6.5, the pH adjustment being made by adding sodium hydroxide (the addition of the copper salt having caused a slight drop in pH).

At each pH, experiments were carried out at two different sequestrant levels:

(i) a sequestrant level of 0.00286 g/l, calculated as equivalent to the concentration in the final rinse when using a detergent composition containing 0.5 wt% of the sequestrant; and

(ii) a sequestrant level of 0.00572 g/l, calculated as equivalent to the concentration in the final rinse when using a detergent composition containing 1.0 wt% of the sequestrant.

Control runs containing no sequestrant, and containing neither copper ions nor sequestrant, were also carried out at each pH. No detergent ingredients were present in these experiments.

Colour changes were monitored as reflectances differences at 620 nm. Two different effects are in operation here: dye fading, which leads to a generally small reflectance increase; and dye darkening (colour degradation) resulting from the presence of the copper ions, leading to a rather larger reflectance decrease. Both effects are undesirable and result in deterioration of the appearance of the fabric. Ideally the reflectance difference observed should be close to zero or a small increase (not greater than 3 units, and preferably not greater than 2 units).

The results were as shown in the Table below.

Example	Sequestrant	Lower sequestrant level (0.5%)		Higher sequestrant level (1.0%)	
		pH 9.5	pH 6.5	pH 9.5	pH 6.5
A	Water alone	+ 2.40	+ 2.49	+ 2.40	+ 2.49
B	Water + Cu^{2+}	- 5.62	-20.00	- 5.62	-20.00
1	IDS	+ 1.04	+ 0.95	+ 2.64	+ 1.22
C	NTA	+ 4.05	+ 3.38	+ 4.95	+ 4.11
D	EDTMP	+ 0.87	-10.23	+ 1.06	- 6.10
E	EDDS	+ 4.43	- 4.35	+ 4.91	+ 3.98
F	STP	- 6.03	-20.90	-15.72	-15.71
G	Mg silicate	- 3.88	-15.40	- 4.18	-13.27

It will be seen that only IDS gave reflectance differences within the 0 to +3 range under all four conditions. EDTMP performed well at pH 9.5 but not at pH 6.5. With NTA, dye fading predominated. This also occurred with EDDS under most conditions. The inorganic sequestrants, sodium tripolyphosphate and magnesium silicate, had only an insignificantly small protective effect against darkening.

EXAMPLE 2 - COMPARATIVE EXAMPLES H TO P

Protection of new white fabrics from discolouration by mixed heavy metal ions

The procedure of Example 1 was repeated at pH 6.5 using white cotton fabrics and a "cocktail" of heavy metal ions:

Cu^{2+}	0.5 ppm
Fe^{3+}	2.5 ppm
Mn^{2+}	2.0 ppm
Zn^{2+}	5.0 ppm

For the white fabrics, discolouration was monitored by means of reflectance changes at 450 nm. For white fabrics there is no colour fading to consider, and the ideal here is for a result as close as possible to zero.

Example	Sequestrant	0.5%	1.0%
H	Water alone	- 0.20	- 0.20
J	Water + ions	- 1.66	- 1.66
2	IDS	- 0.32	- 0.38
K	NTA	- 1.62	- 1.30
L	EDTMP	- 0.54	- 0.54
M	EDDS	- 2.37	- 2.03
N	STP	- 0.89	- 0.71
P	Mg silicate	- 2.23	- 1.71

EXAMPLE 3, COMPARATIVE EXAMPLE Q

Protection of new white silk fabric from colour degradation by mixed heavy metal ions using sequestrants in detergent powder compositions

A detergent powder formulation was prepared by conventional spray-drying and postdosing techniques to the following formulation:

	wt%
Sodium linear alkylbenzene sulphonate (NaLAS)	22.92
Sodium silicate (anhydrous)	4.67
Sodium tripolyphosphate	18.67
Sodium carboxymethyl cellulose	0.25
Polyacrylate polymer	0.70
Calcite	10.00
Sodium sulphate, water, impurities, sequestrant if present	to 100

Comparative Example Q: no sequestrant
Example 3: plus 1.25 wt% IDS

The fabrics were washed five times in a wash liquor comprising 20/6° FH (Ca/Mg) water containing a "cocktail" of heavy metal ions and 2.5 g/litre of the detergent composition, and with the pH adjusted to 9.5 using dilute sulphuric acid. The washes were carried out in tergotometers at a liquor to cloth ratio of 200:1 for 30 minutes at 30°C and 90 rpm. After each wash, two rinses

was carried out at a liquor to cloth ratio of 200:1 and the fabrics were line dried.

The "cocktails" of heavy metal ions were as follows:

	Wash	Rinse
Cu^{2+}	0.5 ppm	0.05 ppm
Fe^{3+}	2.5 ppm	0.1 ppm
Mn^{2+}	2.0 ppm	2.0 ppm
Zn^{2+}	5.0 ppm	1.1 ppm

The results (ΔR at 450 nm) were as follows:

Example	Sequestrant	ΔR (450 nm)	
		after 1 wash	after 5 washes
Q	None	- 1.71	- 2.60
3	IDS	- 1.25	- 1.63

The reduced yellowing of the fabrics washed with the composition of Example 3 was visually apparent.

EXAMPLE 4, COMPARATIVE EXAMPLES R and S

Protection of new white silk from colour degradation by mixed heavy metal ions using sequestrants in detergent powder compositions

The procedure of Example 3 was repeated using the same powder formulation but the pH was adjusted to the lower value of 8.5, and a total of 10 washes per example were carried out. Reflectances were measured after 5 and 10 washes.

The sequestrants used were IDS (invention) and NTA (comparative), at a level of 1.25 wt%.

Example	Sequestrant	ΔR (450 nm)	
		after 5 washes	after 10 washes
R	None	- 0.85	- 1.72
4	IDS	+ 0.42	+ 0.66
S	NTA	- 0.11	- 0.57

EXAMPLE 5, COMPARATIVE EXAMPLE T

Protection of new coloured fabrics from colour degradation by mixed heavy metal ions (2.5 ppm Fe^{3+} and 0.5 ppm Cu^{2+}) using sequestrants in detergent powder compositions

A multi-wash experiment using panellists and handwash conditions was carried out. The colour care benefit was determined using coloured monitors.

A detergent powder composition was prepared by conventional spray-drying and postdosing techniques to the following formulation:

	wt%
Sodium linear alkylbenzene sulphonate (NaLAS)	22.92
Sodium silicate (anhydrous)	5.37
Sodium tripolyphosphate	18.67
Sodium carboxymethyl cellulose	0.25
Polyacrylate polymer	0.70
Calcite	10.00
Sodium sulphate, fluorescer, zeolite, perfume, enzymes, water, impurities, sequestrant if present	to 100

Comparative Example T: no sequestrant
Example 5: IDS at 0.625% of the formulation.

12 wash cycles in total were carried out using the following conditions:

- Trace levels of iron (2.5 ppm) and copper (0.5 ppm)

- Product dosage: 5 g/l (formulation below)
- Water hardness: 10 °FH Ca
- Liquor to cloth ratio in the main wash: 6 to 1
- Liquor to cloth ratio in the rinse: 20 to 1
- Number of rinses: 2
- Rubbing time: 30 seconds per piece.
- Wash temperature: 30 °C.
- Total washload weight: 1 kg.

The coloured monitors were line-dried in the shade.

Colour care benefits were determined both instrumentally (reflectance, ΔE) and by visual assessment.

Instrumental measurements

The reflectance change ΔE, indicative of total colour change across the whole visible spectrum, was measured. The lower the figure, the better the result.

Fabric	ΔE	
	T (no sequestrant)	5 (IDS 0.625%)
Red linen	4.3	3.7
Yellow cotton	7.3	6.2
Deep green cotton	2.0	1.8
Light blue Cotton	7.7	6.8
Lavender cotton	4.3	3.9

Visual assessment by panellists

Visual assessment was carried out by experienced panellists using the universal grey scale [ISO 105-A02: 1993, BS EN 20105-A02: 1995, BS 1006-A02: 1990, Society of Dyers and Colourists Standard Methods 5th Edition A02]:

5 = no colour change from original fabric colour

1 = large change from original fabric colour

Therefore, the higher the visual assessment score, the nearer the test fabric is to the new (unwashed) fabric.

Fabric	Visual assessment (grey scale, 1-5)	
	T No sequestrant	5 IDS 0.625%
Red linen	1.9	2.5
Yellow cotton	2.3	2.8
Deep green cotton	2.9	3.1
Light blue Cotton	2.5	2.9
Lavender cotton	3.1	3.3
Light blue poly-cotton	2.9	3.1

EXAMPLE 6, COMPARATIVE EXAMPLES U to X

Restoration/recovery of coloured fabrics that have been colour-damaged by exposure to copper ions

These experiments demonstrate the benefits of IDS in restoring colour-damaged coloured fabrics.

The fabrics used were cotton dyed with Direct Red 80. They were pre-treated with demineralised water containing 0.5 ppm Cu^{2+} ions and having the pH adjusted to 6.5 by means of sodium hydroxide. The pretreatment was carried out using tergotometers at 30°C, 90 rpm and a liquor to cloth ratio of 200:1, then the fabrics were line dried.

The fabrics were then washed in a wash liquor containing 2.5 g/l of the detergent composition used in Example 3, and 0.5 ppm Cu^{2+}, in 20/6° Ca/Mg French hard water): the pH was adjusted from 9.9 to 9.5 using dilute sulphuric acid. The washes were carried out in tergotometers at 30°C, 90 rpm and a liquor to cloth ratio of 200:1. The washes were followed by two rinses in 20/6° Ca/Mg French hard water containing 0.05 ppm Cu^{2+} at a liquor to cloth ratio of 200:1, and the fabrics were then line dried.

The sequestrants, where present, were dosed directly into the wash liquor in the amounts indicated in the table below (percentages based on the detergent composition).

The procedure was also repeated using 15-minute handwashes (using a strictly controlled protocol) at liquor to cloth ratios of 25:1 and 7:1 instead of the tergotometer washes at 200:1.

Colour changes were monitored by reflectance changes at 620 nm, the standard being the fabrics prior to pretreatment. All values were negative, the ideal being the smallest possible negative value.

The results are shown in the Table below.

Example		200:1 tergo	25:1 handwash	7:1 handwash
U	After pretreatment	-19.93	-19.93	-19.93
V	Detergent composition without sequestrant	-11.98	- 8.51	- 6.86
6	IDS 1.25%	- 3.50	- 2.64	- 0.31
W	NTA 1.71%	- 6.44	- 4.42	- 1.50
X	STP 3.85%	-12.32	- 9.55	- 3.18

Similar results were obtained in a repeat experiment in which the fabrics were not dried between washes.

EXAMPLES 7 and 8, COMPARATIVE EXAMPLES Y and Z

The procedure of Example 6 was repeated at a 200:1 liquor to cloth ratio using both IDS and HIDS, and gave similar results:

Example		200:1 tergo
Y	After pretreatment	-23.68
Z	Detergent composition without sequestrant	-13.40
7	IDS 1.25%	- 5.69
8	HIDS 1.25%	- 5.28

EXAMPLE 9 - COMPARATIVE EXAMPLES AA to DD

Restoration/recovery of coloured fabrics that have been colour-damaged by exposure to mixed heavy metal ions

The procedure of Example 6 was repeated using, instead of copper ions alone, the "cocktails" of heavy metal ions (different for wash and for rinse) used in Example 3. For the pretreatment step the same "cocktail" was used as for the wash.

Similar results to those of Example 6 were obtained, as shown in the Table below.

Example		200:1 tergo	25:1 handwash	7:1 handwash
AA	After pretreatment	-18.08	-18.08	-18.08
BB	Detergent composition without sequestrant	-10.61	-14.83	-14.10
9	IDS 1.25%	- 2.59	- 7.59	- 7.73
CC	NTA 1.71%	- 3.38	- 8.22	-11.50
DD	STP 3.85%	- 7.76	-15.69	-13.11

EXAMPLES 10 TO 16

Restoration/recovery of coloured fabrics that have been colour-damaged by exposure to copper ions, using a wider range of formulations

The procedure of Example 6 was repeated using six different formulations of varying pH. The tergotometer method of previous examples was used. For each formulation, the initial pH was adjusted slightly downwards, using dilute sulphuric acid, to mimic the effect of soil on wash pH.

The washing and rinsing regime was as described in Example 6, and colour changes were monitored as reflectance changes (ΔR) at 620 nm.

The formulations, and the reflectance results, are shown in the following Tables.

Formulations

	10	11	12	13
NaLAS	10.00	22.00	28.00	15.00
NaPAS	15.00			
Nonionic C_{12-15} 7EO				1.00
Na silicate(anhydr)		6.00	8.00	15.00
Na sulphate	19.29	37.90	15.16	23.90
Na tripolyphosphate	30.00	20.00	23.49	
SCMC	0.75	0.70	0.65	0.40
Polymer**	0.50		0.50	
Calcite	10.00		3.64	
Zeolite (78%)				20.00
Citric acid		1.50		
Na carbonate		1.00		
Na bicarbonate			10.00	16.00
Enzyme 2*				0.25
Enzyme 3*	0.53	0.81	0.49	
Enzyme 5*		0.16	0.19	
Water, impurities, sequestrant (if present)	to 100	to 100	to 100	to 100
pH	8.8	9.4	9.6	9.6
adjusted pH	8.6	8.9	9.1	9.1

	14	15	16
NaLAS	22.92	20.00	16.80
Nonionic C_{12-15} 7EO			2.50
Na silicate(anhydr)	5.36	12.00	8.22
Na sulphate	28.71	39.12	20.17
Na tripolyphosphate	18.67	12.00	24.00
SCMC	0.25	0.50	1.00
Polymer**	0.70	0.50	0.50
Calcite	10.00		
Zeolite (anhydr)	1.56		
Zeolite (78%)			4.93
Na carbonate		8.00	15.00
Enzyme 1*	0.16		
Enzyme 4*		0.51	0.56
Water, impurities, sequestrant (if present)	to 100	to 100	to 100
pH	9.9	10.3	10.5
adjusted pH	9.4	9.8	10.0

*Enzyme 1: protease (Purafect 2100G)

*Enzyme 2: protease (Savinase 12T)

*Enzyme 3: protease/lipase/amylase (60/15/15): Savinase/Lipolase/Termamyl

*Enzyme 4: protease/lipase (80/20): Savinase/Lipolase

*Enzyme 5: cellulase: Clazinase 7000G

All specific enzyme names are Trade Marks

**polyacrylate, or acrylate/maleate copolymer

Reflectance results

Ex.	pH		ΔR at 620 nm			
	Initial	Adjusted	After pretreat-ment	No sequest-rant	IDS 1.25%	NTA 1.71%
10	8.8	8.6	-20.31	-8.52	-3.11	-4.37
11	9.4	8.9	-19.97	-8.55	-2.40	-3.59
12	9.6	9.1	-19.99	-7.37	-1.80	-2.30
13	9.6	9.1	-18.50	-4.23	-2.14	-3.30
14	9.9	9.4	-19.72	-7.83	-2.27	-3.52
15	10.3	9.8	-19.27	-4.13	-2.14	-3.30
16	10.5	10.0	-19.50	-3.47	-1.60	-1.92

EXAMPLE 17, COMPARATIVE EXAMPLE EE

Prevention of dye fading from chlorine using sequestrants in detergent powder formulations (chlorine scavenging)

A multi-wash tergotometer experiment was carried out using the detergent composition of Example 3, without sequestrant (Comparative Example EE) or containing 1 wt% of IDS (Example 17). The colour care benefit was determined using a range of coloured cotton monitors.

The wash conditions were as follows:

- Trace levels of metal ions:
  Fe = 2.5 ppm, Cu = 0.5 ppm, Zn = 5 ppm, Mn = 2 ppm
- Trace level of NaOCl: 0-5 ppm
- Product dosage: 2.5 g/l (formulation below)
- Water hardness (wash and rinse): 26 °FH Ca
- Liquor to cloth ratio in the wash and rinse: 200 to 1
- Wash time: 20 minutes
- Number of rinses: 2
- Agitation: 75 rpm
- Wash temperature: 40 °C.

The washed fabrics were line-dried in the shade.

Reflectance results

The lower the figure, the better the result.

Colour of cotton monitor	Wavelength measured	Reflectance change	
		Example EE (No sequestrant)	Example 17 (IDS 1.0%)
Yellow	460 nm	13.59	0.51
Orange	510 nm	3.25	0.72
Navy	620 nm	2.97	0.79

EXAMPLE 18: COMPARATIVE EXAMPLES FF AND GG

Stain removal from fabrics using sequestrants in detergent powder formulations

Stain removal was assessed using a handwash methodology and the following detergent powder formulation:

	wt%
Sodium linear alkylbenzene sulphonate (NaLAS)	23.71
Sodium silicate (anhydrous)	7.37
Sodium tripolyphosphate	18.77
Sodium carbonate	7.00
Sodium carboxymethyl cellulose	0.52
Polyacrylate polymer	0.54
Calcite	9.88
Sodium sulphate, fluorescer, zeolite, perfume, enzymes, water, impurities, sequestrant if present	to 100

Comparative Example FF: no sequestrant
Comparative Example GG: 1 wt% NTA
Example 18: 1 wt% IDS

The wash conditions were as follows:

- Trace levels of metal ions: Fe = 0.1 ppm, Cu = 0.01 ppm, Zn = 0.1 ppm, Mn = 0.2 ppm
- Product dosage: 2 g/l
- Water hardness:
  - Wash: 45 °FH (Ca:Mg 3:1)
  - Rinse: 25 °FH (Ca:Mg 3:1)

- Liquor to cloth ratio in the soak and wash: 7 to 1
- Liquor to cloth ratio in the rinse: 3 to 1
- Soak time: 20 minutes
- Number of rinses: 2
- Rubbing time: 30 seconds per piece
- Wash temperature: 22°C
- Total load weight: ca. 700 g.

The washed fabrics were line-dried in the shade.

Reflectance results (ΔE) were as follows. These represent residual stain and therefore the lower the figure, the better the results. The results on red mud were significant to 95%, while directional improvements were observed on blood and tea.

Example	Sequestrant	ΔE (residual stain)		
		Blood	Tea	Red mud
FF	None	7.84	30.13	19.66
18	IDS	7.20	28.90	14.98
GG	NTA	7.62	30.00	19.86

EXAMPLE 19, COMPARATIVE EXAMPLE HH

Stain removal from fabrics using sequestrants in detergent powder formulations (tergotometer methodology)

Stain removal was also assessed using a tergotometer method, and the formulation of Example 3.

Comparative Example HH contained no sequestrant, while Example 3 contained 1 wt% of IDS.

Wash conditions:

Trace levels of metal ions:
Fe = 2.5 ppm, Cu = 0.5 ppm, Zn = 5 ppm, Mn = 2 ppm

- Product dosage: 2.5 g/l
- Water hardness (wash and rinse): 26 °FH Ca
- Liquor to cloth ratio in the wash and rinse: 200 to 1
- Wash time: 20 minutes
- Number of rinses: 2
- Agitation: 75 rpm
- Wash temperature: 40°C.

The washed fabrics were line-dried in the shade.

Stain removal results are shown below. These are a measure of the stain removed so the larger the figure, the better the result. The improvement here was statistically significant and visually apparent.

Example	Sequestrant	ΔR 580 (Stain removal)
		Red wine (Empa 114)
HH	None	12.67
19	IDS	14.59

EXAMPLES 20 to 22

Concentrated (high bulk density) detergent compositions containing IDS

Phosphate-built and zeolite-built detergent powder formulations of high bulk density (850-900 g/l) containing IDS were prepared to the following formulations:

	20	21	22
NaLAS	15.63	22.82	18.00
Nonionic C_{12-15} 7EO	7.00		3.25
Na sulphate			1.30
Na tripolyphosphate		30.57	
SCMC	0.60	0.77	1.00
Polymer**		1.96	2.00
Zeolite (78%)	44.65	23.13	50.00
Na carbonate	12.12	6.73	19.00
Na bicarbonate	17.77	5.87	
Enzyme 6*	2.00		
Enzyme 7*		0.51	
Enzyme 4			0.45
IDS	1.25	1.25	1.25
Fluorescer, perfume, speckles, water	to 100	to 100	to 100
0.25% solution pH	9.8	10.0	10.6

*Enzyme 6: protease/lipase (72/28): Savinase/Lipolase

*Enzyme 7: protease/cellulase (50/50): Purafect 2100G/Clazinase 7000G

EXAMPLES 23 and 24

Liquid detergent compositions containing IDS

Liquid detergent compositions were prepared to the following formulations:

	23	24
NaLAS	12.50	10.50
Nonionic C_{12-15} 7EO		2.25
SLES	6.66	2.25
Na tripolyphosphate		15.00
Mg sulphate ($7H_2O$)	2.50	
Na tetraborate		4.00
Boric acid	0.50	
Glycerine CP		6.00
Polymer		0.12
Preservatives	0.08	
Blue dye	0.02	
IDS	1.25	1.25
Perfume	0.15	0.40
Water	to 100	to 100
0.25% solution pH	6.5	9.0

EXAMPLE 25

Liquid detergent composition containing IDS

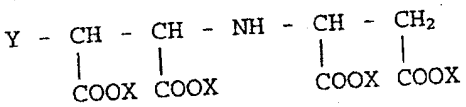
A liquiid detergent composition containing a lower level of IDS was prepared to the following formulation. The IDS was incorporated in liquid form (30% active).

Ingredient	Weight %
Sodium linear alkylbenzene sulphonate	8.655
Nonionic surfactant 7EO	2.885
Zeolite	20.000
Glycerol	4.000
Borax	3.200
Citric acid	2.500
Sodium hydroxide	1.719
IDS (sodium salt)	0.667
Silicone oil/silica	0.200
Sodium xylene sulphonate	0.200
Calcium chloride dihydrate	0.150
Soil release polymer (PET/POET)	0.100
Sodium polyacrylate (Sokalan PA50)	0.100
Protease	0.100
Amylase	0.100
Fluorescer	0.025
Perfume	0.250
Preservative	0.016
Water to 100%	42.378

* * *

CLAIMS

1 A non-bleaching laundry detergent composition comprising surfactant, builder, and optionally other non-bleach detergent ingredients, and also containing from 0.05 to 2.5 wt% of a compound of the formula I:



(I)

wherein Y is H or OH, and X is H or a solubilising cation.

2 A detergent composition as claimed in claim 1, characterised in that it comprises:

(a) from 5 to 60 wt% of one or more detergent surfactants,

(b) from 10 to 80 wt% of one or more detergency builders,

(c) from 0.05 to 2.5 wt% of a compound of the formula I,

(d) optionally other non-bleach detergent ingredients to 100 wt%.

3 A detergent composition as claimed in claim 1 or claim 2, characterised in that it comprises from 0.2 to 2.5 wt% of the compound of the formula I.

4 A detergent composition as claimed in any preceding claim, characterised in that the compound of the formula I is iminodisuccinic acid or a salt, preferably the sodium salt, thereof.

5 A detergent composition as claimed in any preceding claim, characterised in that it has an 0.25% aqueous solution pH (in demineralised water at 30°C) not exceeding 10.5, preferably not exceeding 10.0, more preferably not exceeding 9.5.

6 A detergent composition as claimed in any preceding claim, characterised in that it is in particulate form.

7 A detergent composition as claimed in any one of claims 1 to 5, characterised in that it is in liquid form.

8 A process for laundering white or coloured textile fabrics while maintaining and/or restoring the colour fidelity of the fabrics, which process comprises laundering the fabrics by hand or machine in a wash liquor containing a detergent composition as claimed in claim 1.

9 A process as claimed in claim 8, characterised in that the laundering process is carried out at a wash liquor pH not exceeding 10.5, preferably not exceeding 10.0.

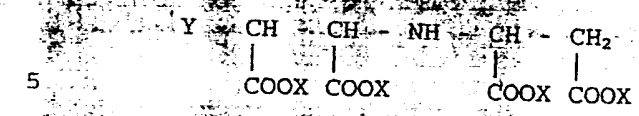
10 A process as claimed in claim 8 or claim 9, characterised in that the laundering process is carried out by hand.

11 A method of protecting new white or coloured textile fabrics from colour degradation on laundering, which comprises laundering the fabrics by hand or machine in a wash liquor containing a detergent composition as claimed in any one of claims 1 to 7.

12 A method of restoring colour fidelity in white or coloured textile fabrics that have been laundered, which comprises laundering the fabrics by hand or machine in a wash liquor containing a detergent composition as claimed in any one of claims 1 to 7.

13 A method of removing stains from textile fabrics, which comprises laundering the fabrics by hand or machine in a wash liquor containing a detergent composition as claimed in any one of claims 1 to 7.

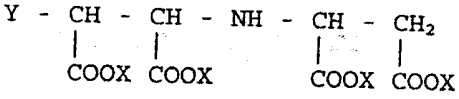
14 Use of a compound of the formula I:



(I)

wherein Y is H or OH, and X is H or a solubilising cation, in an amount of from 0.05 to 2.5 wt%, in a laundry detergent composition to protect new white or coloured textile fabrics from colour degradation on laundering.

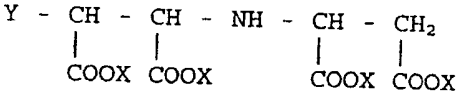
15 Use of a compound of the formula I:



(I)

wherein Y is H or OH, and X is H or a solubilising cation, in an amount of from 0.05 to 2.5 wt%, in a laundry detergent composition to restore colour fidelity in white or coloured textile fabrics that have been laundered.

16 Use of a compound of the formula I:



(I)

wherein Y is H or OH, and X is H or a solubilising cation, in an amount of from 0.05 to 2.5 wt%, in a laundry detergent composition as a chlorine scavenger to prevent the fading of chlorine-sensitive dyes on coloured textile fabrics during laundering.

Use of a compound of the formula I

wherein Y is H or OH, and X is H or a solubilising cation, in an amount of from 0.05 to 2.5 wt%, in a laundry detergent composition to improve its stain removal performance.

INTERNATIONAL SEARCH REPORT

Intern. ...al Application No
PCT/GB 99/04139

IPC ...SIFICATION OF SUBJECT MATTER
...22/29

According to International Patent Classification (IPC) or to both national cla... and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DE 43 11 440 A (HENKEL KGAA) 13 October 1994 (1994-10-13) page 2 -page 3; claims	1-10,12, 13,15
X	GB 474 082 A (A. CARPMAEL) 24 March 1937 (1937-03-24) page 2 -page 4; examples 8,12	1-15,17
X	DE 40 24 552 A (HENKEL KGAA) 6 February 1992 (1992-02-06) page 3; claims; examples	1,3,5,7

-/—

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

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"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
31 March 2000	18/04/2000

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Pfannenstein, H

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INTERNATIONAL SEARCH REPORT

International Application No
PC[illegible]1997[illegible]

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication,where appropriate, of the relevant pas[illegible]	Relevant to claim No.
X	DATABASE WPI Section Ch[illegible]ek 199512 Der[illegible] Publications Ltd., London, GB; Class A25, AN 1995-085720 XP002134500 & JP 07 011294 A (KAO CORP), 13 January 1995 (1995-01-13) abstract	1-6
A	US 3 697 453 A (TATE BRYCE E ET AL) 10 October 1972 (1972-10-10) cited in the application claims; examples IV,VI	1
A	WO 91 17234 A (PROCTER & GAMBLE) 14 November 1991 (1991-11-14) cited in the application page 2 -page 3; claims page 6	16

1

Form PCT/ISA/210 (continuation of second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

Information on patent family members

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